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In re Application of:

PERSSON, M. et al.

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European Patent Application No.: 99850074.8

US Provisional Patent Application No.: 60/132,359:

Priority Date: May 4, 1999

Group Art Unit:

For: SILICA-BASED SOLS

BOX PATENT APPLICATION Assistant Commissioner of Patents Washington, D.C. 20231

PRELIMINARY AMENDMENT

Preliminary to examination, please amend the above-identified application as follows:

IN THE SPECIFICATION:

Page 1, line 1,

Silica-based sols

This application is a continuation of PCT/SE00/00822, filed April 28, 2000, which claims priority of European Patent Application No. 99850074.8, filed May 4, 1999, U.S. Provisional Patent Application No. 60/132,359, filed May 4, 1999, Swedish Patent Application No. 9901687-5, filed May 6, 1999, European Patent Application No. 99850160.5, filed October 29, 1999 and U.S. Provisional Patent Application No. 60/162,445, filed October 29, 1999.

IN THE CLAIMS:

Please amend claim 1 as follows, cancel claims 2-25 without prejudice and add new claims 26-72:

- (Amended) A process for the production of an aqueous sol containing silica-based particles which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO_2 content within the range of from 4.5 to 8% by weight to form an alkalised sol having a pH of at least 7;
- (c) allowing particle growth of the alkalised sol for at least 10 minutes; and
- (d) alkalising the obtained sol to a pH of at least 10.0.
- 26. (New) The process according to claim 1, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to (b).
- 27. (New) The process according to claim 1, wherein the process further comprises:
- (e) concentrating the alkalised sol subjected to particle growth obtained according to (c).
- 28. (New) The process according to claim 1, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to (d).
- 29. (New) The process according to claim 1, wherein the aqueous sol obtained in the process has a specific surface area of at least 90 m^2/g aqueous sol.
- 30. (New) The process according to claim 26, wherein the aqueous sol obtained in the process has a specific surface area of at least 95 m²/g aqueous sol.
- 31. (New) The process according to claim 1, wherein the alkalisation according to (b) and (d) is carried out by means of an aqueous silicate solution.

- 32. (New) The process according to claim 1, wherein the particle growth according to (c) is carried out at a temperature within the range of from 35 to 95°C.
- 33. (New) The process according to claim 1, wherein the alkalisation according to (d) produces a sol having a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1 and a pH of at least 10.6.
- 34. (New) The process according to claim 1, wherein the process further comprises addition of an aluminium-containing compound, a boron-containing compound or a mixture thereof.
- 35. (New) The process according to claim 1, wherein the silica-based particles obtained in the process have a specific surface area of at least 550 m^2/g SiO₂.
- 36. (New) An aqueous sol containing silica-based particles obtained by a process which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol having a pH of at least 7;
- (c) allowing particle growth of the alkalised sol for at least 10 minutes; and
- (d) alkalising the obtained sol to a pH of at least 10.0.
- 37. (New) The aqueous sol according to claim 36, wherein the process further comprises:
- (e) concentrating the sol obtained according to (c).
- 38. (New) The aqueous sol according to claim 36, wherein the process further comprises:
- (e) concentrating the sol obtained according to (d).

- 39. (New) The aqueous sol according to claim 37, wherein it has a specific surface area of at least $95 \text{ m}^2/\text{g}$ aqueous sol.
- 40. (New) The aqueous sol according to claim 36, wherein the aqueous sol has a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1 and a pH of at least 10.6.
- 41. (New) The aqueous sol according to claim 36, wherein the sol comprises an aluminum-containing compound, a boron-containing compound or a mixture thereof.
- 42. (New) The aqueous sol according to claim 36, wherein the silica-based particles have a specific surface area of at least $550 \text{ m}^2/\text{g SiO}_2$.
- 43. (New) A process for the production of an aqueous sol containing silicabased particles which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol;
- (c) heat-treating the alkalised sol at a temperature of at least 30°C; and
- (d) alkalising the heat-treated sol to a pH of at least 10.0.
- 44. (New) The process according to claim 43, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to step (b).
- 45. (New) The process according to claim 43, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to step (c).
- 46. (New) The process according to claim 43, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to step (d).

- 47. (New) The process according to claim 43, wherein the aqueous sol obtained in the process has a specific surface area of at least 90 m²/g aqueous sol.
- 48. (New) The process according to claim 43, wherein the aqueous sol obtained in the process has a specific surface area of at least 95 m²/g aqueous sol.
- 49. (New) The process according to claim 43, wherein the alkalisation according to (b) and (d) is carried out by means of an aqueous silicate solution.
- 50. (New) The process according to claim 43, wherein the heat-treatment according to (c) is carried out for 20 to 240 minutes.
- 51. (New) The process according to claim 43, wherein the alkalisation according to (d) produces a sol having a molar ratio of SiO₂ to M₂O, where M is alkali metal or ammonium, within the range of from 15:1 to 30:1 and a pH of at least 10.6.
- 52. (New) The process according to claim 43, wherein the process further comprises addition of an aluminum-containing compound, a boron-containing compound or a mixture thereof.
- 53. (New) The process according to claim 43, wherein the silica-based particles obtained in the process have a specific surface area of at least 550 m²/g SiO₂.
- 54. (New) An aqueous sol containing silica-based particles obtained by a process comprising:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol;
- (c) heat-treating the alkalised sol at a temperature of at least 30°C; and
- (d) alkalising the heat-treated sol to a pH of at least 10.0.

- 55. (New) The aqueous sol according to claim 54, wherein the process further comprises:
- (e) concentrating the sol obtained according to (c).
- 56. (New) The aqueous sol according to claim 54, wherein the process further comprises:
- (e) concentrating the sol obtained according to (d).
- 57. (New) The aqueous sol according to claim 54, wherein it has a specific surface area of at least 95 m²/g aqueous sol.
- 58. (New) The aqueous sol according to claim 54, wherein it has a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1 and a pH of at least 10.6.
- 59. (New) The aqueous sol according to claim 54, wherein it comprises an aluminum-containing compound, a boron-containing compound or a mixture thereof.
- 60. (New) The aqueous sol according to claim 54, wherein the silica-based particles have a specific surface area of at least 550 m²/g SiO₂.
- 61. (New) A process for the production of an aqueous sol containing silicabased particles which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol;
- (c) heat-treating the alkalised sol at a temperature within the range of from 35 to 95°C for 20 to 240 minutes;
- (d) alkalising the heat-treated sol to a pH of at least 10.0 and a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1;
- (e) concentrating the sol obtained according to (c) or (d); and

- (f) providing an aqueous sol which has a specific surface area of at least 95 m²/g aqueous sol and contains silica-based particles which have a specific surface area of at least 550 m²/g SiO₂.
- 62. (New) The process according to claim 61, wherein the alkalisation according to step (b) and step (d) is carried out by means of an aqueous silicate solution.
- 63. (New) The process according to claim 61, wherein the alkalisation according to (d) produces a pH of at least 10.6.
- 64. (New) An aqueous sol containing silica-based particles obtained by a process which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol;
- (c) heat-treating the alkalised sol at a temperature within the range of from 35 to 95°C for 20 to 240 minutes:
- (d) alkalising the heat-treated sol to a pH of at least 10.0 and a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1;
- (e) concentrating the sol obtained according to step (c) or step (d); and
- (f) providing an aqueous sol which has a specific surface area of at least 95 m 2 /g aqueous sol and contains silica-based particles which have a specific surface area of at least 550 m 2 /g SiO $_2$.
- 65. (New) The aqueous sol according to claim 64, wherein it has a pH of at least 10.6.
- 66. (New) An aqueous sol containing silica-based particles, said sol has a specific surface area of at least 115 m²/g aqueous sol and said silica-based particles have a specific surface area of at least 550 m²/g SiO₂ and less than 1000 m²/g SiO₂.

- 67. (New) The aqueous sol according to claim 66, wherein the sol has a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 40:1.
- 68. (New) The aqueous sol according to claim 66, wherein the sol has an S-value within the range of from 25 to 35%.
- 69. (New) An aqueous sol containing silica-based particles, wherein the sol has a specific surface area of at least $115 \text{ m}^2/\text{g}$ aqueous sol and an S-value within the range of from 10 to 45%.
- 70. (New) The aqueous sol according to claim 69, wherein the sol has a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 40:1.
- 71. (New) The aqueous sol according to claim 69, wherein the silica-based particles have a specific surface area of at least 550 m²/g SiO₂.
 - 72. (New) An aqueous silica-based sol having:
- (a) a specific surface area of at least 115 m²/g aqueous sol;
- (b) an S-value within the range of from 10 to 45%; and
- (c) a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 40:1;

and containing

(d) silica-based particles which have a specific surface area of at least 550 and less than 1000 m²/g SiO₂.

IN THE ABSTRACT:

Please add the following abstract on a separate page following the claims:

Abstract of the Disclosure

A process for the production of an aqueous sol containing silica-based particles which comprises (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol; (b) alkalising the acid sol at an SiO2 content within the range of from 4.5 to 8% by weight; (c) allowing particle growth of the alkalised sol for at least 10 minutes; or heat-treating the alkalised sol at a temperature of a least 30°C; (d) alkalising the obtained sol to a pH of at least 10.0; and (e) optionally concentrating the sol obtained according to (b), (c) or (d) to provide an aqueous sol containing silica-based particles and having a specific surface area of at least 90 m²/g aqueous sol; as well as an aqueous sol containing silica-based particles obtainable by the process. The invention also relates to an aqueous sol containing silica-based particles which sol has a specific surface area of at least 115 m²/g aqueous sol and an S-value within the range of from 10 to 45% or contains silica-based particles having a specific surface area of at least 550 and less than 1000 m²/g SiO₂. The invention further relates to the use of the aqueous sol containing silica-based particles as a drainage and retention aid in the production of paper as well as a process for the production of paper from an aqueous suspension containing cellulosic fibres, and optional filler, in which silicabased particles and at least one charged organic polymer are added to the cellulosic suspension.

Remarks

This preliminary amendment amends the specification, claims and abstract, and adds new claims without the addition of new matter thereby. A marked version showing the amendments is attached hereto. Presently, claims 1 and 26-72 are pending.

Early and favorable consideration of this application is respectfully requested.

Respectfully submitted,

Lainie E. Parker

Attorney for Applicants Registration No.: 36,123

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MARKED VERSION

IN THE SPECIFICATION:

Page 1, after line 1 (the title), insert, --This application is a continuation of PCT/SE00/00822, filed April 28, 2000, which claims priority of European Patent Application No. 99850074.8, filed May 4, 1999, U.S. Provisional Patent Application No. 60/132,359, filed May 4, 1999, Swedish Patent Application No. 9901687-5, filed May 6, 1999, European Patent Application No. 99850160.5, filed October 29, 1999 and U.S. Provisional Patent Application No. 60/162,445, filed October 29, 1999.--

IN THE CLAIMS:

Please amend claim 1 as follows, cancel claims 2-25 without prejudice and add new claims 26-72:

- 1. (Amended) [Process] <u>A process</u> for the production of an aqueous sol containing silica-based particles which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol having a pH of at least 7;
- (c) allowing particle growth of the alkalised sol for at least 10 minutes; and
- (d) alkalising the obtained sol to a pH of at least 10.0[; and
- (e) optionally concentrating the sol obtained according to (b), (c) or (d) to provide an aqueous sol containing silica-based particles and having a specific surface area of at least 90 m²/g aqueous sol].
- -26. (New) The process according to claim 1, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to (b).
- 27. (New) The process according to claim 1, wherein the process further comprises:
- (e) concentrating the alkalised sol subjected to particle growth obtained according to (c).

- 28. (New) The process according to claim 1, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to (d).
- 29. (New) The process according to claim 1, wherein the aqueous sol obtained in the process has a specific surface area of at least 90 m²/g aqueous sol.
- 30. (New) The process according to claim 26, wherein the aqueous sol obtained in the process has a specific surface area of at least 95 m²/g aqueous sol.
- 31. (New) The process according to claim 1, wherein the alkalisation according to (b) and (d) is carried out by means of an aqueous silicate solution.
- 32. (New) The process according to claim 1, wherein the particle growth according to (c) is carried out at a temperature within the range of from 35 to 95°C.
- 33. (New) The process according to claim 1, wherein the alkalisation according to (d) produces a sol having a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1 and a pH of at least 10.6.
- 34. (New) The process according to claim 1, wherein the process further comprises addition of an aluminium-containing compound, a boron-containing compound or a mixture thereof.
- 35. (New) The process according to claim 1, wherein the silica-based particles obtained in the process have a specific surface area of at least 550 m²/g SiO₂.
- 36. (New) An aqueous sol containing silica-based particles obtained by a process which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol having a pH of at least 7;

- (c) allowing particle growth of the alkalised sol for at least 10 minutes; and
- (d) alkalising the obtained sol to a pH of at least 10.0.
- 37. (New) The aqueous sol according to claim 36, wherein the process further comprises:
- (e) concentrating the sol obtained according to (c).
- 38. (New) The aqueous sol according to claim 36, wherein the process further comprises:
- (e) concentrating the sol obtained according to (d).
- 39. (New) The aqueous sol according to claim 37, wherein it has a specific surface area of at least 95 m²/g aqueous sol.
- 40. (New) The aqueous sol according to claim 36, wherein the aqueous sol has a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1 and a pH of at least 10.6.
- 41. (New) The aqueous sol according to claim 36, wherein the sol comprises an aluminum-containing compound, a boron-containing compound or a mixture thereof.
- 42. (New) The aqueous sol according to claim 36, wherein the silica-based particles have a specific surface area of at least 550 m²/g SiO₂.
- 43. (New) A process for the production of an aqueous sol containing silicabased particles which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol;
- (c) heat-treating the alkalised sol at a temperature of at least 30°C; and
- (d) alkalising the heat-treated sol to a pH of at least 10.0.

- 44. (New) The process according to claim 43, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to step (b).
- 45. (New) The process according to claim 43, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to step (c).
- 46. (New) The process according to claim 43, wherein the process further comprises:
- (e) concentrating the alkalised sol obtained according to step (d).
- 47. (New) The process according to claim 43, wherein the aqueous sol obtained in the process has a specific surface area of at least 90 m²/g aqueous sol.
- 48. (New) The process according to claim 43, wherein the aqueous sol obtained in the process has a specific surface area of at least 95 m²/g aqueous sol.
- 49. (New) The process according to claim 43, wherein the alkalisation according to (b) and (d) is carried out by means of an aqueous silicate solution.
- 50. (New) The process according to claim 43, wherein the heat-treatment according to (c) is carried out for 20 to 240 minutes.
- 51. (New) The process according to claim 43, wherein the alkalisation according to (d) produces a sol having a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1 and a pH of at least 10.6.
- 52. (New) The process according to claim 43, wherein the process further comprises addition of an aluminum-containing compound, a boron-containing compound or a mixture thereof.

- 53. (New) The process according to claim 43, wherein the silica-based particles obtained in the process have a specific surface area of at least 550 m²/g SiO₂.
- 54. (New) An aqueous sol containing silica-based particles obtained by a process comprising:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol;
- (c) heat-treating the alkalised sol at a temperature of at least 30°C; and
- (d) alkalising the heat-treated sol to a pH of at least 10.0.
- 55. (New) The aqueous sol according to claim 54, wherein the process further comprises:
- (e) concentrating the sol obtained according to (c).
- 56. (New) The aqueous sol according to claim 54, wherein the process further comprises:
- (e) concentrating the sol obtained according to (d).
- 57. (New) The aqueous sol according to claim 54, wherein it has a specific surface area of at least 95 m²/g aqueous sol.
- 58. (New) The aqueous sol according to claim 54, wherein it has a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1 and a pH of at least 10.6.
- 59. (New) The aqueous sol according to claim 54, wherein it comprises an aluminum-containing compound, a boron-containing compound or a mixture thereof.
- 60. (New) The aqueous sol according to claim 54, wherein the silica-based particles have a specific surface area of at least 550 m²/g SiO₂.

- 61. (New) A process for the production of an aqueous sol containing silicabased particles which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol;
- (c) heat-treating the alkalised sol at a temperature within the range of from 35 to 95°C for 20 to 240 minutes;
- (d) alkalising the heat-treated sol to a pH of at least 10.0 and a molar ratio of SiO_2 to M_2O_1 , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1;
- (e) concentrating the sol obtained according to (c) or (d); and
- (f) providing an aqueous sol which has a specific surface area of at least 95 m²/g aqueous sol and contains silica-based particles which have a specific surface area of at least 550 m²/g SiO₂.
- 62. (New) The process according to claim 61, wherein the alkalisation according to step (b) and step (d) is carried out by means of an aqueous silicate solution.
- 63. (New) The process according to claim 61, wherein the alkalisation according to (d) produces a pH of at least 10.6.
- 64. (New) An aqueous sol containing silica-based particles obtained by a process which comprises:
- (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol;
- (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight to form an alkalised sol;
- (c) heat-treating the alkalised sol at a temperature within the range of from 35 to 95°C for 20 to 240 minutes;
- (d) alkalising the heat-treated sol to a pH of at least 10.0 and a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1;
- (e) concentrating the sol obtained according to step (c) or step (d); and

- (f) providing an aqueous sol which has a specific surface area of at least 95 m 2 /g aqueous sol and contains silica-based particles which have a specific surface area of at least 550 m 2 /g SiO $_2$.
- 65. (New) The aqueous sol according to claim 64, wherein it has a pH of at least 10.6.
- 66. (New) An aqueous sol containing silica-based particles, said sol has a specific surface area of at least 115 m 2 /g aqueous sol and said silica-based particles have a specific surface area of at least 550 m 2 /g SiO $_2$ and less than 1000 m 2 /g SiO $_2$.
- 67. (New) The aqueous sol according to claim 66, wherein the sol has a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 40:1.
- 68. (New) The aqueous sol according to claim 66, wherein the sol has an S-value within the range of from 25 to 35%.
- 69. (New) An aqueous sol containing silica-based particles, wherein the sol has a specific surface area of at least 115 m²/g aqueous sol and an S-value within the range of from 10 to 45%.
- 70. (New) The aqueous sol according to claim 69, wherein the sol has a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 40:1.
- 71. (New) The aqueous sol according to claim 69, wherein the silica-based particles have a specific surface area of at least 550 m²/g SiO₂.
 - 72. (New) An aqueous silica-based sol having:
- (a) a specific surface area of at least 115 m²/g aqueous sol;
- (b) an S-value within the range of from 10 to 45%; and

(c) a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 40:1;

and containing

(d) silica-based particles which have a specific surface area of at least 550 and less than $1000 \text{ m}^2/\text{g SiO}_2$. - -

IN THE ABSTRACT:

Please add the following abstract on a separate page following the claims:

- - Abstract of the Disclosure

A process for the production of an aqueous sol containing silica-based particles which comprises (a) acidifying an aqueous silicate solution to a pH of from 1 to 4 to form an acid sol; (b) alkalising the acid sol at an SiO₂ content within the range of from 4.5 to 8% by weight; (c) allowing particle growth of the alkalised sol for at least 10 minutes; or heat-treating the alkalised sol at a temperature of a least 30°C; (d) alkalising the obtained sol to a pH of at least 10.0; and (e) optionally concentrating the sol obtained according to (b), (c) or (d) to provide an aqueous sol containing silica-based particles and having a specific surface area of at least 90 m²/g agueous sol; as well as an aqueous sol containing silica-based particles obtainable by the process. The invention also relates to an aqueous sol containing silica-based particles which sol has a specific surface area of at least 115 m²/g aqueous sol and an S-value within the range of from 10 to 45% or contains silica-based particles having a specific surface area of at least 550 and less than 1000 m²/g SiO₂. The invention further relates to the use of the aqueous sol containing silica-based particles as a drainage and retention aid in the production of paper as well as a process for the production of paper from an aqueous suspension containing cellulosic fibres, and optional filler, in which silicabased particles and at least one charged organic polymer are added to the cellulosic suspension. - -